

REGISTERED LETTER

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Ns. rif. SL:vs/M2-2562 February 25, 2005  
Our ref:

Vs. rif. APV43301EP  
Your ref:

**Attn. Mr. Anthony P. Venturino**  
STEVENS, DAVIS, MILLER & MOSHER, L.L.P.  
1615 L Street, N.W. Suite 850  
WASHINGTON, DC 20036  
U.S.A.

**Re: European Patent Application No. 00932781.8 in the name of RHODIA Inc.**

Dear Mr. Venturino,



We would like to inform you that we have received the second Communication pursuant to Article 96(2) EPC (Office Action, copy attached) issued by the Examining Division of the European Patent Office in relation to the above-identified patent application.

As you can see, after the amendments to the claims filed with the reply to the first Office Action (our letter of September 28, 2004 to the EPO), the Examiner has carried out a new search and discovered two new documents, GB-A-2,027,045 (D5) and US-A-4,542,175 (D6), copy of which is attached hereto. According to the Examiner's opinion, both D5 and D6 anticipate the subject-matter of the amended claim 1.

As remarked in our previous communications on this case (our e-mails of August 25 and September 27, 2004), in order to overcome the objections raised in this second Office Action, our reply must clearly indicate what the respective distinguishing process and product features are and what problem is solved by such distinguishing features in a non-obvious way.

Therefore, we kindly ask to you to obtain by the inventors a detailed report on said distinguishing features of the patent application and on the differences between the latter and the subject-matter of the prior art documents cited.

The Examiner has also remarked that the deemed lack of novelty of the independent product claim referring to the polymers could determine a possible lack of unity of the numerous independent composition and use claims (so-called lack of unity *a posteriori*).

Please take into account also this possible problem when re-drafting the claims in order to overcome the objections raised by the European Examiner

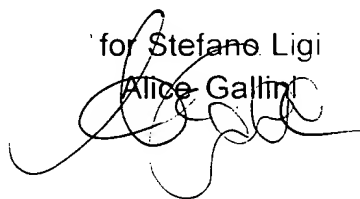
Should you need any kind of further clarifications or explanations, please contact us promptly. In any case, **please provide us with your comments and instructions by no later than April 30, 2005**, so as to be able to prepare and file a reply to the outstanding Office Action within the deadline, i.e. June 3, 2005.

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At your complete disposal for any additional clarification which you may require in this respect, we thank you for your collaboration and remain

Yours faithfully,

for Stefano Ligi  
Alice Gallini



Encl.: - copy of 2nd Office Action issued by the EPO  
- copy of D5 and D6



Bescheid/Protokoll (Anlage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

Datum  
Date 03.02.2005  
Date

Blatt  
Sheet 1  
Feuille

Anmelde-Nr..  
Application No.: 00 932 781.8  
Demande n°

The examination is being carried out on the **following application documents**:

#### Description, Pages

1-149 as published

#### Claims, Numbers

1-23 received on 02.10.2004 with letter of 28.09.2004

The following documents (D) are cited by the examiner (see the Guidelines, C-VI, 8.7). Copies of the documents are annexed to the communication and the numbering will be adhered to in the rest of the procedure:

D5: GB-A-2 027 045

D6: US-A-4 542 175

In his answer to the communication, dated 22.03.04, the applicant introduced original claim 16 into claim 1.

D5, e.g. examples 1-3, claims 1,3, 6 describes copolymers comprising e.g. dimethyl-aminoethyl and acrylate or methacrylate and hydroxypropyl acrylate and (meth)acrylic acid.

See also D6, column 10, line 18 - 21.

The citations anticipate the subject matter now claimed.

As to inventive step it is noted that D5 refers to the same technical as the application.

Should the applicant see a possibility to file new claims that refer to novel and inventive matter the following is recalled:

It is required to exactly specify for any amendments the basis in the description (Art. 123(2) EPC).

In the answer the applicant should identify the respective distinguishing process and product feature (Art. 33(2) PCT), and he should make plausible which technical problem is solved by the distinguishing feature (only by that feature and not in



Bescheld/Protokoll (Ariage)

Communication/Minutes (Annex)

Notification/Procès-verbal (Annexe)

Datum  
Date 03.02.2005  
Date

Blatt  
Sheet 2  
Feuille

Anmelde-Nr.:  
Application No.: 00 932 781.8  
Demande n°:

combination with other known features) in a non obvious way (Art. 54 EPC).

The independent claims should be drafted in the two part form with the distinguishing feature forming the characterising portion (Rule 29(1)EPC).

Also the following still requires attention.

Lack of novelty of the product claims referring to polymers has as consequence that the numerous independent composition and use claims no longer are linked by a novel and inventive concept. Thus, unity of invention is lacking apparently (Art. 82 EPC).

(12) UK Patent Application (19) GB (11) 2 027 045 A

(21) Application No 7926312  
(22) Date of filing 27 Jul 1979  
(23) Claims filed 27 Jul 1979  
(30) Priority data  
(31) 2832944  
(32) 27 Jul 1978  
(33) Fed. Rep. of Germany (DE)  
(43) Application published  
13 Feb 1980  
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C11D 3/37  
(52) Domestic classification  
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C3W 209 211 212 301  
C3Y B101 B230 G140  
C5D 6B12B1 6B12F2  
SB13 6C8  
(56) Documents cited  
None  
(58) Field of search  
C3M  
C3V  
C5D  
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(54) Polymer Solutions Useful for Treating Hair

- (57) The solutions comprise:  
a) 20 to 99% by weight of water;  
b) 0.5 to 79% by weight of a water-soluble cationic vinyl copolymer composed of  
i) monomer units containing a quaternary ammonium salt group;  
ii) monomer units of an  $\alpha, \beta$ -unsaturated carboxylic acid or a water-soluble salt thereof;  
iii) if desired, monomer units containing an amino or primary, secondary, tertiary or quaternary ammonium groups; and  
(iv) also if desired, units of one or more non-ionic comonomers which are copolymerisable with monomers i), ii) and iii);  
the said copolymer containing the

following proportions of monomer units i), ii), and (where present) iii) and iv), namely, at least 5 molar % of monomer units i); more than 20 molar % of monomer units i) and where present, iii); monomer units ii) in an amount of at least 5 molar % but less than the sum of the molar % amounts of monomer units i) and iii); and less than 75 molar % of monomer units iv); and

c) 0.5 to 79% of at least one water-soluble ionic or ionisable compound which is capable of forming a precipitate when admixed with an aqueous solution of poly-(methacryloxyethyltrimethylammonium chloride).

The solutions may be used for example as hair shampoos. The presence in the vinyl copolymer of the specified amount of monomer units i) overcomes previous stability problems encountered with solutions of cationic polymers, which solutions also contain anionic surface active agents or anionic polyelectrolytes.

GB 2 027 045 A

## SPECIFICATION

### Polymer Solutions

The invention relates to aqueous solutions of vinyl copolymers having a cationic character and containing dissolved anionic surface active agents or anionic polyelectrolytes.

5 As a rule, the above-mentioned aqueous solutions are unstable since insoluble salts or complexes 5 are formed from the above-mentioned cationic and anionic components. Thus, for example, water-soluble polymers of quaternised aminoalkyl esters of acrylic or methacrylic acid are precipitated by anionic surface active agents or by polyacrylic acid or alkali metal salts thereof. Nevertheless, it is frequently desired to prepare aqueous solutions containing incompatible components of opposite 10 charge, for example because it is desired to use both components simultaneously in view of their specific functions or because the one component is already present in an aqueous solution and can only be removed with difficulty or not at all before the other component is added. 10

One example of the simultaneous requirement for incompatible counter-ionic constituents is illustrated by cosmetic compositions for the hair. These compositions desirably contain both anionic 15 surface active agents as hair-washing agents and also cationic vinyl polymers as hair strengthening and hair conditioning agents. 15

According to Dutch Patent Application 7607314, stable solutions of this type may be prepared from a mixture of anionic, non-ionic and amphoteric surface active agents with a nitrogen-containing amphoteric polyelectrolyte containing aminoalkyl groups and small quantities of carboxyl groups. 20 However, in practice, the use of anionic surface active agents instead of the mixture composed of three 20 different types of surface active agent is generally preferred.

Stable aqueous solutions of anionic polyelectrolytes and vinyl copolymers with units of unsaturated carboxylic acids, on the one hand, and units with primary, secondary or tertiary amino groups, on the other hand, can be prepared according to U.S. Patent Specification No. 3,950,296 at 25 specific pH values. However, upon adjustment of the pH towards the isoelectric point, insoluble 25 precipitates may occur.

It is an object of the present invention to provide stable aqueous solutions which contain, in solution, vinyl copolymers having a cationic character together with ionic or ionisable compounds, also in solution, which form precipitates in aqueous solutions of poly(meth-acryloxyethyl 30 trimethylammonium chloride), the stability of such solutions being substantially independent of the choice of specific components or the observance of narrow pH ranges. Poly(methacryloxyethyl trimethylammonium chloride) with a mean molecular weight of 100,000 is selected as reference compound for determination of incompatibility in view of its ready availability on an industrial scale. The ionic or ionisable compounds which are incompatible therewith are also incompatible with other 35 cationic polyelectrolytes. By vinyl copolymers with a cationic character are meant those copolymers wherein the stoichiometric proportion of cationic groups, i.e. amino or ammonium groups, clearly predominates over anionic groups, i.e. carboxyl or carboxylate groups. 35

According to the present invention we provide aqueous solutions comprising

- a) 20 to 99% by weight of water;
- 40 b) 0.5 to 79% by weight of a water-soluble cationic vinyl copolymer composed of 40
  - i) monomer units containing a quaternary ammonium salt group;
  - ii) monomer units of an  $\alpha,\beta$ -unsaturated carboxylic acid or a water-soluble salt thereof;
  - iii) if desired, monomer units containing an amino or primary, secondary, tertiary or quaternary ammonium group; and
- 45 iv) also if desired, units of one or more further nonionic comonomers which are copolymerisable 45 with monomers i), ii), and iii); the said copolymer containing the following proportions of monomer units i), ii), and when present iii) and iv), namely, at least 5 molar % of monomers units i); more than 20 molar % of monomer units i) and, when present, iii); monomer units ii) in an amount of at least 5 molar % but less than the sum of the molar % amounts of 50 monomer units i) and, when present, iii); and less than 75 molar % of monomer units iv); and 50
- c) 0.5 to 79% of at least one water-soluble ionic or ionisable compound which is capable of forming a precipitate when admixed with an aqueous solution of poly(methacryloxyethyl-trimethylammonium chloride).

An important factor in the stability of the solutions according to the invention is the content of 55 quaternary ammonium groups in the vinyl copolymer (b). 55

There is not in every case unlimited compatibility between the ionic compounds, but improved compatibility in comparison with purely cationic polyelectrolytes is generally achieved. Improved compatibility may for example be obtained at specific temperatures, concentrations or pH values. There are other instances in which compatibility problems of cationic polyelectrolytes with counter-ionic 60 compounds arise only under certain unfavourable conditions. In these cases, the range of incompatibility within the meaning of the invention can be narrowed. 60

The solutions according to the invention contain in dissolved form the above-mentioned constituents b) and c). In addition thereto, the solutions may contain, depending on the requirements for the intended use, further dissolved or undissolved constituents.

- The vinyl copolymer forming the component b) is composed of vinyl monomers, i.e. monomers with a radically polymerisable or at least copolymerisable carbon double bond in the form of a vinyl group, a vinylidene group or a vinylene group. The vinyl copolymers according to the invention may be composed exclusively of the monomer units i) and ii) and contain in each case more than 10 basic molar % of these units. The cationic groups need not be present entirely in the form of quaternary ammonium groups. They may even be present for the most part in the form of primary, secondary or tertiary amino groups or the primary, secondary or tertiary ammonium groups formed therefrom by salt formation with an acid, provided that the quaternary ammonium groups amount to at least 5 molar % of the monomer units of the vinyl copolymer (b). Preferably, their proportion is 5 to 25 molar %. The cationic character of the vinyl copolymer is ensured as long as the anionic units ii) containing carboxyl or carboxylate groups are present in a stoichiometrically lower amount than the cationic units i) and iii). In the preferred case, the stoichiometric excess of the cationic groups over the anionic groups is 5 to 20 molar %. The proportion of non-ionic monomer units iv) is preferably below 50 molar %.
- Monomer units iii) are preferably derived from vinyl monomers containing a primary, secondary or tertiary amino group, the latter being preferred on account of their good polymerisability. A preferred group of monomers consists of aminoalkyl esters and the aminoalkyl amides of acrylic and methacrylic acid, such as dimethyl-aminoethyl acrylate and methacrylate, diethylaminoethyl acrylate and methacrylate, butylaminoethyl acrylate and methacrylate, piperidinoethyl, morpholinoethyl and piperazinoethyl esters of acrylic and methacrylic acid, N-dimethylaminomethylacrylamide and -methacrylamide and N-dimethylaminopropylacrylamide and -methacrylamide. Further suitable monomers containing amino groups are vinyl-pyridine, vinyl-imidazole and vinyl-imidazoline. The corresponding ammonium salts are obtained from these monomers by reaction with an inorganic or organic acid such as hydrochloric acid, sulphuric acid, acetic acid or chloroacetic acid.
- Monomer units i) containing quaternary ammonium groups are derived from monomers which may in turn be obtained from the above-mentioned monomers containing tertiary amino groups by quaternisation, preferably with methyl chloride, benzyl chloride or dimethyl sulphate.
- Anionic monomer units ii) may be derived, for example from maleic acid, fumaric acid, itaconic acid, crotonic acid and preferably from acrylic or methacrylic acid. The water-soluble salts of these acids, especially the alkali and ammonium salts may also be employed.
- The monomer units iv) optionally present in the copolymer are not ionic or ionisable. These units may be derived from water-soluble monomers, for example from acrylamide and methacrylamide, vinyl pyrrolidone, hydroxyalkylesters of  $\alpha,\beta$ -unsaturated, polymerisable monocarboxylic or dicarboxylic acids, especially those with 2 to 4 carbon atoms in the hydroxyalkyl radical, as well as from the hypothetical monomer vinyl alcohol. The monomer units iv) may also be derived from water-insoluble comonomers in such a quantity that the water-solubility of the polymer is maintained. Said comonomers include the alkyl esters (especially those of  $C_{1-4}$  alcohols) and nitriles of  $\alpha,\beta$ -unsaturated carboxylic acids, vinyl esters of fatty acids, vinyl halides, vinyl aromatics,  $\alpha$ -olefins and/or dienes.
- Especially preferred for hair-cosmetic preparations are those cationic vinyl copolymers in which the stoichiometric ratio of cationic groups i) and iii) to anionic groups ii) is about 1:1, especially in the range between 1:1 and 1.5:1. Typical compositions contain about equal proportions of units ii) and iii) and a relatively small proportion of units i).
- Various processes may be employed to prepare vinyl copolymer b). In addition to processes involving the polymerisation of relatively dilute aqueous monomer solutions in the presence of water-soluble radical-formers, particularly preferred processes include those in which highly concentrated aqueous monomer solutions are polymerised in the presence of water-soluble radical formers or ultra-violet light. Hard polymers are thereby obtained which can be handled as solid products and which are dissolved before use in water to the desired concentration. Polymerisation may be effected for example according to Belgian Patent Specification 695,342 in foil bags, according to German Patent Specification 1,770,588 in open tanks, according to German Offenlegungsschrift 2,545,290 on an endless belt, according to German Offenlegungsschrift 2,009,218 as bead polymerisation in a non-aqueous phase or according to German Offenlegungsschrift 2,322,883 as suspension polymerisation in oil with the formation of a stable organosol soluble in water. The molecular weight of the vinyl copolymer (b) may lie between 1,000 and 1,000,000. They are however preferably prepared in the presence of controllers and have molecular weights between 1,000 and 50,000.
- The compound designated (c) forms with purely cationic polyelectrolytes such as poly(methacryloxyethyl trimethylammonium chloride), water-insoluble salts or complexes which precipitate out of aqueous solution. The precipitating action may manifest itself in the form of clouding, flocculation or the formation of larger insoluble aggregates, for example as sediment. Occasionally, these precipitations occur only at certain temperatures, concentrations or pH values. The precipitating action originates from the anionic component of the compound (c). The compound (c) is a compound composed of anions and cations which dissociates partly or completely in aqueous solution or is converted in aqueous solution into a dissociated compound such as polycarboxylic acids which form by dissociation protons and poly-carboxylate anions. The anions present in the aqueous solution then have a precipitating action on purely cationic polyelectrolytes, if they contain a plurality of charges or are bound to a large hydrophobic radical, i.e. represent anionic surface active agents. With regard to

the anionic surface active agents the solutions may contain conventional soaps as well as all conventional surface active agents of the sulphate and sulphonate type, for example the alkali metal salts of aromatic sulphonic acids, sulphated oils, sulphates of fatty alcohols and the sulphated oxalkylation products of phenols, alkylated phenols, fatty alcohols and fatty acid amides.

- 5 Water-soluble, high-molecular compounds with carboxyl or carboxylate groups which are not compatible with purely cationic polymers, but are compatible with the vinyl polymers (b) of the cationic type according to the invention include polyacrylic acid, partly saponified polyacrylonitrile or polyacrylamide, polymethacrylic acid, copolymers of maleic acid with ethylene or styrene and carboxymethyl cellulose, particularly those compounds with molecular weights above 1,000. The same  
10 applies to the water-soluble salts, especially the alkali salts and ammonium salts of these compounds. 10

For example, polyacrylic acid and poly(dimethylaminoethyl methacrylate) are incompatible with one another in aqueous solution. In contrast, concentrated aqueous solutions (30% by weight of solid) may be prepared comprising polyacrylic acid and a copolymer with a cationic character which is composed of

- 15 37 molar % of dimethylaminoethyl methacrylate, 15  
6 molar % of methacryloxyethyl trimethyl ammonium chloride,  
37 molar % of methacrylic acid,  
20 molar % of hydroxypropyl acrylate.

- 20 With a stoichiometric ratio of cationic groups of the copolymer to the carboxyl groups of the polyacrylic acid in the range from 2:1 to over 7:1 these solutions are fully compatible and clear Diluted solutions (1% by weight of solid) are compatible and clear at least at values over 4:1 for the above-mentioned ionic ratio. 20

- 25 The concentration of the oppositely charged compounds b) and c) in the solutions according to the invention may reach the saturation limit. Above a solid content of 80% by weight, liquid systems cannot generally be obtained. For high concentrations of the vinyl copolymer-b) the solutions are high-viscosity. Of particular industrial interest is the concentration range of from 0.5 to 15% by weight for each of the compounds b) and c), since here the problems of compatibility are generally most important. When the total concentration falls below a value of 1% by weight, indications of incompatibility gradually disappear, so that in this range there is usually no need for application of the present invention. 30

- Aggravated problems of compatibility usually arise when anionic compounds c) are present in more than a stoichiometric quantity with regard to a cationic polyelectrolyte. The invention is therefore of particular importance for aqueous solutions in which the ionic or ionisable compound c) is present, calculated in ionic charge equivalents, in a stoichiometrically larger quantity than the amino and ammonium groups in the vinyl copolymer b). 35

The following Examples illustrate the present invention.

#### Example 1

Hair shampoos stable in storage are prepared from:

- 40 a) 10 parts by weight (C<sub>12</sub>—C<sub>14</sub>)-n-alkyl polyethyleneglycolether sulphate sodium salt,  
b) 5 parts by weight of vinyl copolymer A to F with a cationic character. 40

For comparison, the same quantity of the purely cationic poly(methacryloxyethyl trimethylammonium chloride (Molecular weight 100,000) is used instead of the vinyl copolymers. Flakes arise spontaneously and are gradually deposited.

#### Polymers A to F

- 45 Compositions in Molar Parts: 45

Polymer	A	B	C	D	E	F	
Methacrylic acid	47	40	44.2	27.9	35	24.8	
Dimethylaminoethyl methacrylate	47	40	44.2	27.9	35	24.8	
50 Methacryloxyethyl trimethylammonium chloride	6	20	6.2	20.2	5.8	5.8	50
2-hydroxypropyl acrylate	—	—	5.4	24.0	—	20.6	
55 Ethyl acrylate	—	—	—	—	12.1	12.0	55
Methyl methacrylate	—	—	—	—	12.1	12.0	

#### Example 2

An aqueous solution is prepared from the following constituents:

- 60 5 parts by weight of a copolymer consisting of  
45% by weight of methacryloxyethyl trimethylammonium chloride  
5% by weight of methacrylic acid 60



- 5% by weight of dimethylaminoethyl methacrylate  
 45% by weight of 2-hydroxypropyl acrylate  
 10 parts by weight of (C<sub>12</sub>—C<sub>14</sub>)-n-alkyl-polyethylene glycolether sulphate sodium salt  
 85 parts by weight of water.  
 5 The solution was clear and stable. 5

### Example 3

- An aqueous solution is prepared from the following constituents:  
 5 parts by weight of a copolymer consisting of  
 15% by weight of methacryloxyethyl trimethylammonium chloride  
 10% by weight of methacrylic acid  
 10% by weight of dimethylaminoethyl methacrylate  
 65% by weight of 2-hydroxypropyl acrylate  
 10 parts by weight of (C<sub>12</sub>—C<sub>14</sub>)-n-alkyl-polyethylene glycolether sulphate sodium salt  
 85 parts by weight of water.  
 15 The solution was clear and stable. 15

### Claims

1. Aqueous solutions comprising:
  - a) 20 to 99% by weight of water;
  - b) 0.5 to 79% by weight of a water-soluble cationic vinyl copolymer composed of
    - i) monomer units containing a quaternary ammonium salt group;
    - ii) monomer units of an  $\alpha,\beta$ -unsaturated carboxylic acid or a water-soluble salt thereof;
    - iii) if desired, monomer units containing an amino or primary, secondary, tertiary or quaternary ammonium group; and
    - iv) also if desired, units of one or more further non-ionic comonomers which are copolymerisable with monomers i), ii) and iii);
- 25 the said copolymer containing the following proportions of monomer units i), ii), and (where present) iii) and iv), namely, at least 5 molar % of monomer units i); more than 20 molar % of monomer units i) and, where present, iii); monomer units ii) in an amount of at least 5 molar % but less than the sum of the molar % amounts of monomer units i) and, where present, (iii); and less than 75 molar % of monomer units iv); and
  - c) 0.5 to 79% of at least one water-soluble ionic or ionisable compound which is capable of forming a precipitate when admixed with an aqueous solution of poly(methacryloxyethyltrimethylammonium chloride).
- 30 2. Solutions as claimed in claim 1, wherein monomer units iii) of the said vinyl copolymer, when present, are selected from units of aminoalkyl esters and aminoalkyl amides of acrylic and methacrylic acid. 35
3. Solutions as claimed in claim 2, wherein monomer units iii) of the said vinyl copolymer, when present, are selected from units of dimethylaminoethyl acrylate and methacrylate, diethylaminoethyl acrylate and methacrylate, butylaminoethyl acrylate and methacrylate, piperidinoethyl,
  - morpholinoethyl and piperazinoethyl esters of acrylic and methacrylic acid, N-dimethylamino-methyl-acrylamide and -methacrylamide and N-dimethylaminopropylacrylamide and -methacrylamide.
- 40 4. Solutions as claimed in any of the preceding claims, wherein monomer units i) of the said vinyl copolymer are selected from units formed by quaternisation of monomer units iii) as defined in claims 2 and 3.
5. Solutions as claimed in any of the preceding claims, wherein monomer units ii) of the said vinyl copolymer are selected from units of acrylic and methacrylic acids and water-soluble salts thereof. 45
6. Solutions as claimed in any of the preceding claims, wherein monomer units iv) of the said vinyl copolymer, when present, are selected from units of acrylamide and methacrylamide; vinyl-pyrrolidone; hydroxyalkyl esters of  $\alpha,\beta$ -unsaturated, polymerisable mono- and dicarboxylic acids; and vinyl alcohol. 50
7. Solutions as claimed in any of the preceding claims, wherein monomer units i) and, where present, iii) are present in a stoichiometric excess of 5 to 20 molar % over the monomer units ii).
8. Solutions as claimed in any of the preceding claims, wherein the said vinyl copolymer has a molecular weight of 1,000 and 50,000.
- 55 9. Solutions as claimed in any of the preceding claims, wherein the said compound c) comprises an anionic surface active agent. 55
10. Solutions as claimed in any of the preceding claims, wherein the said compound c) comprises a polycarboxylic acid or an anionic polyelectrolyte.
11. Solutions as claimed in any of the preceding claims, wherein the said compound c) is present

in a stoichiometric excess (based on ionic equivalents) over monomers unit(s) i) and, when present, iii) in the said vinyl copolymer.

12. Solutions as claimed in any of the preceding claims, containing 0.5 to 15% by weight of each of b) and c).

5 13. Hair-treatment compositions comprising a solution as claimed in any of the preceding claims. 5

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25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.